<table>
<tr><td>LLNL
Environmental Restoration
Division Standard Operating
Procedure</td><td>TITLE:
Soil Surface Flux Monitoring
of Gaseous Emission</td></tr>
<tr><td>APPROVAL   Date

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ERD SOP-1.11

REVISION: 0

EFFECTIVE DATE:
December 1, 1995

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</table>

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1.0 PURPOSE

This procedure describes the use of emission isolation flux chambers. Emission isolation flux chamber measurements are used to quantify the rate of loss of contaminants, such as volatile organic compounds (VOCs), from soil and ground water to the atmosphere.

2.0 APPLICABILITY

Emission isolation flux chamber data are used to plan remediation of contaminated sites and to monitor the effectiveness of remediation efforts. Emission rates calculated from these data may be used together with exposure models to estimate human health effects.

3.0 REFERENCES

3.1 Kienbusch, M. (1986), *Measurement of Gaseous Emissions Rates from Land Surfaces Using an Emission Isolation Flux Chamber*, User's Guide, EPA Users Guide, Contract No. 68-02-03389-WA18 (EPA/600/8-86/008).

3.2 Eklund, B. (1983), "Practical Guidance for Flux Chamber Measurements of Fugitive Volatile Organic Emission Rates," J. Air Waste Management Assoc., 42:1583-1591.

3.3 U.S. EPA (1989), *Risk Assessment Guidance for Superfund, Vol. I: Human Health Evaluation Manual, Part A, Interim Final*, Office of Emergency and Remedial Response, U.S. Environmental Protection Agency, Washington, D.C. (EPA/540/1-89/002).

4.0 DEFINITIONS

4.1 Collocated Samples

Collocated samples are independent samples collected in such a manner that they are equally representative of the parameter(s) of interest at a given point in space and time.

4.2 Data Logger

A data logger is used to convert the analog signals of various types of traducers to digital format and to log these data in memory with the associated times of measurement. This digitized data may then be retrieved and "downloaded" to a computer for further analysis. Most data loggers can process raw data into values expressed in engineering units.

4.3 Equipment Blank

An equipment blank is an air sample from a flux chamber placed over an inert surface that has come to equilibrium with analyte-free sweep air. Equipment blanks provide a measure of contamination that may have been introduced to the data set by the emission isolation flux chamber system. They are also useful for documenting adequate decontamination of sampling equipment and qualifying data in accordance with Reference 3.3.

4.4 Flux Chamber

An emission isolation flux chamber is an enclosure of known volume with an open end of known area that is placed on soil surfaces to quantify fugitive soil emissions. The chamber contains several penetrations that are used to introduce "sweep-air", to allow chamber-air to exit and for temperature and pressure sensors. Attachment A shows a Flux Chamber schematic.

4.5 ppb

Parts per billion (ppb) as used in this document is a unit of concentration in a gas based on a volume to volume ratio. Ppb refers to the volume of pollutant or compound per billion volumes of air (1 ppb = 1×10^{-9} m^3 analyte/m^3 air).

4.6 ppm

Parts per million (ppm) as used in this document is a unit of concentration in a gas based on a volume to volume ratio. Ppm refers to the volume of pollutants or compound per million volumes of air (1 ppm = 1×10^{-6} m^3 analyte/m^3 air).

4.7 Replicate Sample

Replicate samples are three samples that have been taken sequentially from the same flux chamber under similar conditions. Replicated samples are taken in the field to provide statistical support for the process and to verify the reproducibility of the sampling and analytical processes.

4.8 Soil Surface Flux

Soil surface flux is the rate of exchange of one or more gases between soils and the atmosphere. An emission isolation flux chamber is used to measure this rate by placing the chamber on a soil surface and purging the air inside the chamber with pure sweep-air at a known rate. Gases which diffuse from the soil surface enter the emission isolation flux chamber and mix with the sweep air. By measuring the concentration in the chamber of each target gas once the chamber is at equilibrium, the flux rate ($\mu g \bullet m^{-2} \bullet min^{-1}$), may be calculated with the following formula:

$$F = (SR*C)/A$$

where

F = Flux rate ($\mu g \bullet m^{-2} \bullet min^{-1}$),

SR = Sweep Rate ($m^3 \bullet min^{-1}$),

C = Concentration of analyte ($\mu g \bullet m^{-3}$), and

A = Basal area of the flux chamber (m^2).

4.9 Sweep Air

Sweep air is contaminant free air used to exchange or “sweep” the air inside the flux chamber at a known rate. Sweep air may be provided in the form of ultra-pure “zero air” pressurized gas cylinders with a pressure regulator, or in the form of pumped ambient air filtered through activated carbon and desiccant columns. Accurate measurement of the sweep flow rate is essential to calculate soil vapor flux.

4.10 Thermistor

A thermistor is a thermal resistance device used to measure temperatures. The electrical resistance across this device changes in proportion with changes in temperature. A properly calibrated thermistor can report temperatures with resolutions of less that 0.1^{o} C. Thermistors are typically used in field applications because of their ruggedness, ease of measurement and immunity to electrical noise.

4.11 Thermocouple

A thermocouple is an electronic device used to measure temperatures and is based on a small current that flows through the junction of two dissimilar metals proportional to the temperature of the junction. The output from a thermocouple is only 1 or 2 millivolts. Signals this small are difficult to measure accurately and are subject to any type of electronic noise in the vicinity. When measuring temperature using these devices, extreme care must be taken to compensate for currents generated by temperature at the junction between the thermocouple wire and the data logger.

4.12 Transducers

Transducers are devices that are used to measure some physical parameter, such as pressure or temperature, and convert these measurements to an analog electrical signal.

4.13 Trip Blank

The trip blank is an unused sampling container, such as an evacuated sampling sphere (SUMMA Canister) or an unexposed sorbent cartridge that has been treated in the same way as the containers used for sampling. Trip blanks will indicate any contamination from routine handling practices or from analytical procedures.

4.14 Volatile Organic Compounds (VOCs)

VOCs are frequently found as soil contaminants in industrial areas as a result of their use as solvents and degreasers.

5.0 RESPONSIBILITIES

5.1 Data Management Group (DMG)

The DMG's responsibilities are to receive and process data according to applicable procedures and assist the task leader to develop sampling plans and sampling location IDs.

5.2 Division Leader

The Division Leader's responsibility is to ensure that all activities performed by ERD at the Livermore Site and Site 300 are performed safely and comply with all pertinent regulations and procedures, and provide the necessary equipment and resources to accomplish the tasks described in this procedure.

5.3 Task Leader

The task leader's responsibilities are to write the sampling plan, oversee the field team, deliver all data to the DMG, review the data collected, and prepare any final reports.

5.4 Field Team

The field team is responsible for assembling and testing supplies and equipment prior to work in the field. The field team is responsible for field record-keeping and for collecting all samples according to the sampling plan and this SOP. The field team may make changes to the sampling plan in the field if required by logistics or terrain when authorized by the Task Leader.

6.0 PROCEDURE

6.1 The Sampling Plan

6.1.1 The sampling plan shall contain a site map with sampling areas and locations clearly marked. Each location will be assigned an area designation and location number that will be used on CoCs to identify samples taken at that location.

6.1.2 The number of samples and sampling locations will be selected based on sampling objectives, previous site characterization, ERD Data Quality Objectives (DQOs), and the sampling methodologies recommended in Reference 3.1. Field blanks and equipment blanks will be taken at times and locations specified by the sampling plan in order to meet or exceed these DQOs.

6.1.3 Modifications to the sampling plan are permitted in the field during sampling operations if past history, data anomalies or logistical problems make this necessary. These decisions are made by the person designated by the task leader.

6.2 Office Preparation

6.2.1 The day before flux measurements are made, the internal batteries in the flux chamber controller box, the data logger unit, and the laptop computer must be charged to ensure that enough power is available to perform the flux measurements and record all relevant data.

6.2.2 Prior to departure for the field site, the following supplies must be assembled:

A. Log Book.

B. Appropriate field forms such as Soil Surface Flux Log Sheets (Attachment B) and CoC Forms.

C. Flux chamber controller box with charged internal battery.

D. Flux chamber.

E. Cylinder of compressed zero-air or columns of activated carbon and desiccant for ambient air treatment.

F. Air-pressure regulator or ambient air pump.

G. Data logger(s) equipped with ambient air temperature sensor. If data logger requires programming, be sure to set the time (Pacific Standard Time) and date, and download sampling program before departing to the field.

H. Clean tubing and connections for the analyte-free air supply.

I. Laptop computer with charged internal battery and a sufficient number of charged external batteries to last over the sampling period.

6.3 Field Preparations

6.3.1 Position the flux chamber on the substrate at the sample location. The rim of the flux chamber should be worked into the surface 2–3 cm. to minimize ambient air intrusion.

6.3.2 Secure the zero-air cylinder to a stake or other upright structure with a strap. Connect the air-pressure regulator to the cylinder and to the “Sweep In” port of chamber controller box using 1/4-in. flexible tubing with a quick-disconnect fitting.

6.3.3 Place the chamber controller on a level surface and adjust the Magnahelic to read 0.5 inches H_2O (the indicator will be centered and vertical).

6.3.4 Connect the “Sweep Out” port of the chamber controller box to the central port on the flux chamber using 1/4-in. tubing with a quick-disconnect fitting. Using 1/4-in. tubing, connect the “Return” port on the controller to the lateral port of the flux chamber with no internal protrusions. Connect the “Mag” (Magnahelic) port on the controller to a lateral port of the flux chamber with no internal protrusions.

6.3.5 Connect the 4-pin electrical plug from the chamber to the “Fan” port of chamber controller. Connect the “Sensor Out” port on the controller box to the data logger.

6.4 Operations

6.4.1 Startup procedures

A. Place the controller box switches labeled “Fan” and “Pump” in the “On” position. Turn on the zero-air cylinder and adjust the controller box “Sweep Air” rotometer to a setting that will give a flow of about 3.2 L/minute. Adjust the “Return Air” rotometer to a setting that will also give a flow of about 3.2 L/minute. Fine tune this setting, as required, so that the net pressure drop between the atmosphere and the chamber is zero (Magnahelic reading of 0.5).

B. Maintain these conditions for at least 30 minutes, allowing the chamber has come to equilibrium with VOCs diffusing from the soil.

6.4.2 Sampling procedures

A. Attach the sampling device to the peripheral port on the flux chamber which is connected to the stainless steel tube leading to a position within the chamber below the fan. Enter the sample number, and the serial number of the sampling device (SUMMA canister or sorbent cartridge) on the data logging form.

B. Log sampling conditions (i.e., chamber temperature and sample device condition, such as the vacuum of a SUMMA Canister) and the start time on the data logging form. Begin sampling by filling canister or passing known volume of air through sorbent trap. Since any type of sampling device will withdraw air from the chamber, it is necessary to compensate for this by reducing the flow rate of the “Return Air” rotometer on the controller box by the sampling flow rate. Log the “Sweep Air” and “Return Air” flow rates on the data logging form.

C. After a sample has been taken, log the stop time, final flow rates, sample volume or SUMMA canister vacuum and chamber temperature on the data logging form.

D. Enter the sample number and other requisite information on the CoC form. Label the sampling device (SUMMA canister or sorbent cartridge) with the sample number, data and time.

6.4.3 Equipment Blanks

A. One equipment blank is usually taken at the beginning of the day and at the conclusion of sampling for each flux chamber.

B. Place the flux chamber on a contaminate-free stainless steel surface. Teflon has a tendency to absorb, then release VOCs to which it is exposed. Therefore, Teflon sheets should not be used for this purpose, unless the user can guarantee that the Teflon is not off-gassing VOCs. The stainless steel sheets may be baked-out from time to time to remove any residues.

C. Set up the equipment as described in Sections 6.2 and 6.3.1. Collect sample as described in Section 6.3.2.

6.4.4 Trip Blanks

A. Select unused sampling device(s) and designate it/them as a trip blank(s) as described in the sampling plan (at least one per day).

B. Attach a sample identification number the trip blank as described in the sampling plan, and enter this information on CoC.

C. Send the trip blank to the contract analytical laboratory with other samples taken that day.

6.4.5 Collocated Samples

Collocated samples will be collected at the frequency indicated by the sampling plan and usually depends on the sampling media. For canisters, 10% are usually collected as collocated samples. For sorbent cartridges, replicate samples at each location are usually collected.

6.5 Post Operation

6.5.1 Handle and ship all samples according to SOP 4.4, “Guide to Handling, Packaging, and Shipping of Samples.” SUMMA canisters do not need to be placed in coolers and have no preservation requirement. However, SUMMA canisters should be shipped to the analytical laboratory for analysis within 24 h.

6.5.2 If a data logger was used to record chamber and ambient temperature and chamber differential pressure, download these data to a computer and save to a file so that they will be available for use in the flux calculations.

6.6 Data Analysis

6.6.1 When analytical results are available, the concentrations of each analyte should be converted into $\mu g \bullet m^{-3}$. If the analytical laboratory reports the concentration of each analyte as ppb(v/v) or ppm (v/v), convert the concentration to $\mu g/m^3$ using the following equations:

C = ppb * (analyte’s molecular weight in grams)/24.4 or,

$C = (ppm * 10^3) * (\text{analyte's molecular weight in grams})/24.4.$

Where 24.4 L/mole is the number of liters per mole of air (L/mole) at 2˚C.

The flux rate, F, ($\mu g \bullet m^{-2} \bullet min^{-1}$) may then be calculated for each analyte by using the following equation:

$$F = SR*(C / SV) /A$$

where

SR = Sweep rate ($m^3 \bullet min^{-1}$),

C = Total $\mu g \bullet m^{-3}$ of analyte, and

A = Basal area of the flux chamber.

6.6.2 Compare the equipment and/or trip blank sample concentrations to the concentrations of the same chemicals detected in the samples. In accordance with Reference 3.3, soil flux measurements from individual analyses with a VOC concentration less than five times the maximum VOC concentration detected in any equipment and/or trip blank sample are treated as nondetections. However, all sampling should be conducted to minimize any detection in blank samples.

6.7 Clean Up

6.7.1 If the results of the post-sampling equipment blank reveal unacceptable levels of analyte residue in the chamber or support equipment, the following procedure should be used to decontaminate the equipment.

A. Disassemble the chamber and wash all interior surfaces with 0.1% Triton X-100 in de-ionized water. Rinse or wipe all parts three times with solvent-free distilled water. Air dry all parts in a clean environment and reassemble the unit.

B. Replace all air-line tubing as needed to prevent leakage or to eliminate contamination.

7.0 QA RECORDS

7.1 Chain-of-Custody Forms

7.2 Logbooks

7.3 Sampling and Analysis Plans

7.4 Soil Surface Flux Log Sheet

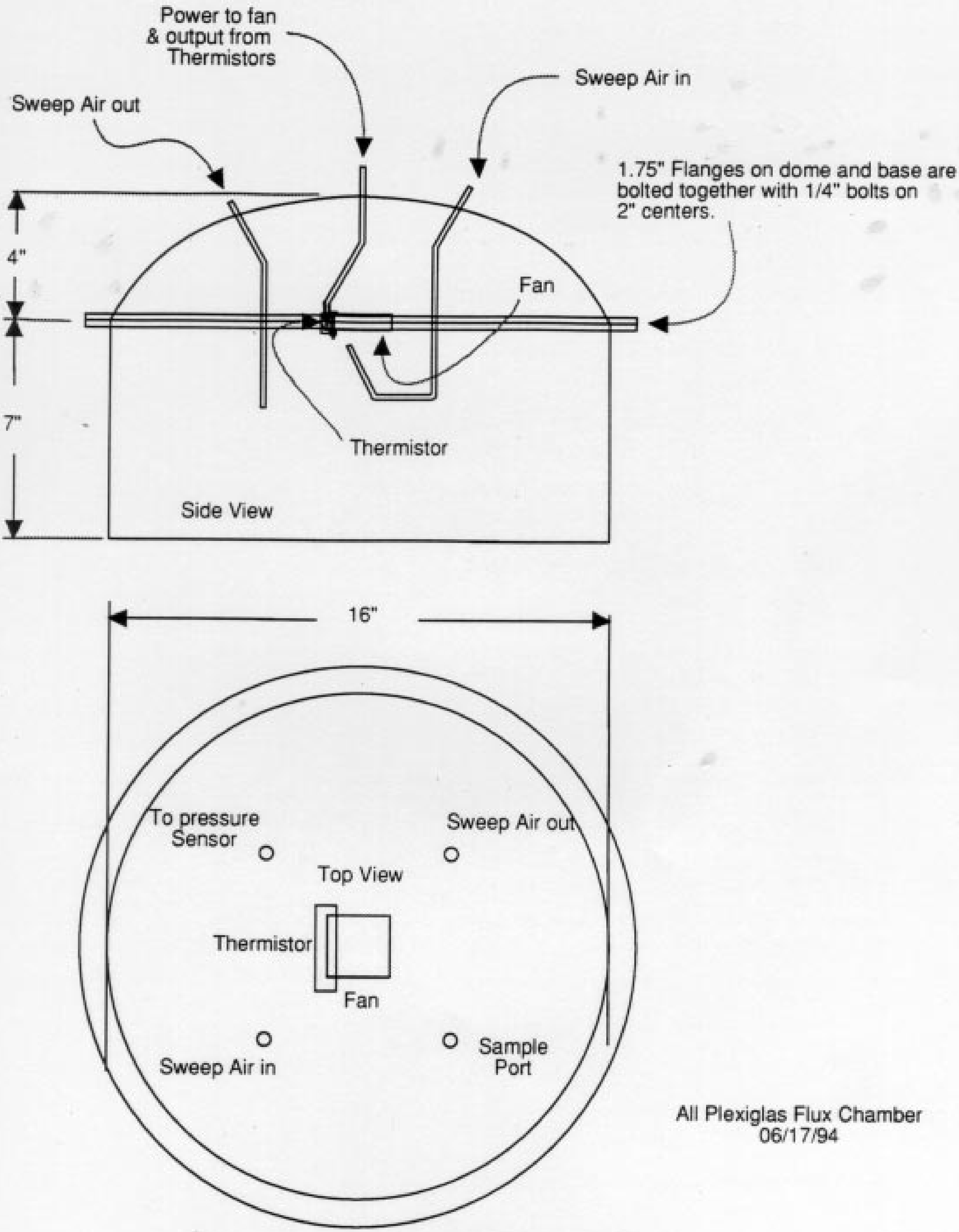
8.0 ATTACHMENTS

Attachment A—Schematic of Flux Chamber

Attachment B—Soil Surface Flux Log Sheet

Attachment A

Schematic of Flux Chamber



All Plexiglas Flux Chamber
06/17/94

Attachment B

Soil Surface Flux Log Sheet

Soil Surface Flux Log Sheet

Soil Surface Flux Log Sheet #__________

Site Number: Date: Chamber:

Purge Start: Sweep In: Sweep Out: Air Temp:
Sample Start: Sweep In: Sweep Out: Ch. Temp:
#______ Stop: Sample Volume: Tube/Canister ID:
Notes: Initials:
Sample Start: Sweep In: Sweep Out: Ch. Temp:
#______ Stop: Sample Volume: Tube/Canister ID:
Notes: Initials:
Sample Start: Sweep In: Sweep Out: Ch. Temp:
#______ Stop: Sample Volume: Tube/Canister ID:
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Sample Start: Sweep In: Sweep Out: Ch. Temp:
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